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Application No: GB 0004661.5 **Examiner:** Stephen Smith
Claims searched: 1-28 **Date of search:** 24 May 2000

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): B8C(CF12)

Int Cl (Ed.7): B65B 31/00, 31/02, 31/04, 63/00, 63/08

Other: ONLINE:EPODOC, WPI

Documents considered to be relevant:

Category	Identity of document and relevant passage		Relevant to claims
X	GB 1543512	(LIOT) lines 59-82 of page 1; line 116 of page 2 to line 35 of page 3	1-4, 7, 9, 13-15, 20, 21, 25, 26

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

1 543 512

PATENT SPECIFICATION

(11) 1 543 512

(21) Application No. 42879/77 (22) Filed 14 Oct. 1977
(31) Convention Application No. 76012
(32) Filed 15 Oct. 1976 in
(33) Luxembourg (LU)
(44) Complete Specification published 4 April 1979
(51) INT CL² A23B 5/00
(52) Index at acceptance A2D 2E1 2E2 2J 2L 2X6 3A3

(19)

(54) LIQUID EGG PRODUCTS

(71) We, R. LIOT SA, a French Company, of 134—144 Avenue Laferriere, 9400 Creteil, France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to the preservation of liquid egg products based on whole eggs, egg white or egg yolk, in concentrated or diluted form, the osmotic pressure of which has been increased by the addition of foodstuffs.

Numerous patents which relate to the preservation of egg products are known, particularly French Patent No. 679,991 involving an evaporation treatment in vacuo, and French Patent No. 1,271,154 involving the sterilisation of the egg product other than by raising the temperature, for example by an ultra-violet ray treatment.

However, either these processes do not impart true sterilisation to the egg product, or the sterilisation obtained involves too significant a denaturation of the liquids, glucides and proteins in the egg.

With regard to the process described in French Patent No. 679,991, in which sugar is added to the liquid egg product before its pasteurisation, the effects of heat are accelerated by evaporation in vacuo, even at very low temperature; in fact, it is well known that degradation of the proteins by heat increases very rapidly when the heating is carried out under reduced pressure. It is also known that simple pasteurisation of the egg products over a few minutes at atmospheric pressure involves significant denaturation of the constituents of the egg, if the temperature of the treatment reaches about 54° C for the egg white, 60° C for the whole egg, and 65° C for the egg yolk, under given pasteurisation conditions.

We have found unexpectedly that liquid egg products for prolonged preservation can be obtained at low cost when a controlled and sufficient deoxygenation of the liquid egg product is carried out, which product is concentrated or non-concentrated, contains salt or sugar or both.

A liquid egg product for prolonged preservation can thus be obtained, which exhibits practically no denaturation of the proteins and the preservation of which can be prolonged for several months without changing the organoleptic properties of the product or, for example, its emulsifying or swelling capacity.

According to the process of the present invention:

(a) salt in an amount of at least 5% by weight based on the weight of the final product, or sugar in an amount of at least 30% and preferably 35% by weight based on the weight of the final product, or both salt and sugar, is added to a concentrated or un-concentrated initial liquid egg product until an osmotic pressure of 20 and preferably 25 atmospheres is obtained, to provide a homogeneous mixture;

(b) dissolved gases are removed from the egg product until the oxygen content is less than 3 ppm and preferably less than 1 ppm (by weight) based on the weight of the final egg product, it being possible for stages (a) and (b) to be carried out in either order;

(c) optionally the product obtained after stages (a) and (b) is heated to a temperature which is lower than the coagulation temperature; and

(d) optionally the product is placed in an airtight packaging under a high vacuum or in the presence of a gas which is inert to foodstuffs, other than carbon dioxide gas, the temperature during the process never exceeding the coagulation temperature of the egg product so that coagulation does not take place and no denaturation of the proteins takes place. All parts in this Specification are by weight unless otherwise stated, based on the weight of the final product.

As the initial liquid egg product, whole egg, egg yolk, or egg white may be used; it may come directly from freshly cracked hens' or other birds' eggs; the dissolved oxygen content a few hours after cracking the eggs is generally 5 to 10 ppm. This oxygen content results in part from leaving the unbroken

eggs in the air; an egg contains an average of 3.4 ppm of dissolved oxygen 2 to 3 days after collecting.

The salt and sugar, as well as the other usual additives such as benzoates or colourants like carotene, which may be used, are introduced in stage (a) of the process although such other additives can be added later.

The term "sugar" is used herein to denote saccharose, galactose and analogous sugars, preferably non-fermentable sugars having a comparable osmotic pressure.

The deoxygenation can be carried out in a variety of ways, on the non-concentrated or concentrated liquid egg product, by a discontinuous or continuous process, by a process using an inert entraining gas, preferably not carbon dioxide, with or without recycling, or by subjecting the egg product to reduced pressure or to heating to, say, 45° to 75° C, especially 50° to 65° C, for a short time, or by a combination of these processes, O_2 and CO_2 being purged in known manner when recycling is used. This treatment is in no way comparable with a pasteurisation process. The temperature and duration of the heating depend to some extent on the desired nature of the egg product but they should always be chosen so as to prevent denaturation and/or coagulation of the egg, particularly when the process is carried out in a partial vacuum. When a concentrated product is used as the starting product, the concentration process can be carried out by any method provided that in the case of a heat treatment the above-mentioned conditions are observed, such that the temperature preferably does not exceed 50° C at atmospheric pressure for a time not exceeding 6 hours.

The initial liquid egg product in the non-concentrated state coming from fresh eggs generally contains dissolved oxygen in an amount of about 10 ppm in the white, 6 to 8 ppm in the yolk and 7 to 8 ppm for the egg as a whole. Carbon dioxide can be present not only in the form of dissolved gas but also chemically bound as carbonate and bicarbonate, giving rise to an equilibrium. In the initial liquid egg product the content of carbon dioxide which can be liberated, including dissolved gas and also carbon dioxide in the form of carbonate and bicarbonate, expressed in millimols of CO_2 per millilitre of product is about 30 to 40 millimols for the egg white, 1 to 2 millimols for the yolk and 20 to 24 millimols for the egg as a whole. Clearly the degasification of oxygen requires different conditions than those for carbon dioxide.

We have found that in the presence of a carbon dioxide atmosphere carbon dioxide can be formed from enzymes which are capable of forming carbon dioxide at the expense of saccharose and glucose which results in swelling of the packaging, a modification of the pH, a de-sugaring of the liquid egg product which as a result develops an acetone odour. For all these reasons, therefore, it is preferable not to use carbon dioxide gas as the inert entraining gas.

When egg white is used as the initial egg product the amounts of salt and sugar (when used alone) should be as specified above. When egg yolk is used, the salt if used alone, must be present in an amount of at least 5% and preferably 7% by weight, and the sugar, if used alone, must be present in an amount of at least 35% and preferably 40% by weight. When whole egg is used, it is preferable to use proportions which are close to those used for egg white.

When an egg product containing both salt and sugar is used, the salt is preferably present in an amount of at least 0.5% by weight and the sugar preferably in an amount of at least 25% by weight relative to the weight of the final product, the osmotic pressure being at least 20 atmospheres and preferably 25 atmospheres.

The salt and sugar can be introduced at any time during the treatment, it being possible for the egg product to be concentrated before, during, or after the degassing treatment.

When a non-concentrated liquid egg product is used, the degassing is carried out either directly on the initial liquid product or after incorporation into the product of the additives, that is to say the salt (sodium chloride) and/or the sugar, the whole mixture being perfectly homogenised. Although the introduction of the additives and the degassing can be carried out in any order, the additives are preferably introduced as a first stage into the liquid egg product and, after having obtained a homogeneous mixture, e.g. by stirring, the degassing operation is carried out.

When it is desired to obtain a concentrated final product, it is preferable first to carry out the concentration, for example by ultrafiltration, and then carry out the degassing before or after the introduction of the additives. The solids content should preferably not exceed 60% by weight.

Deoxygenation can be carried out simply by bubbling the inert gas through the liquid egg product. It is also possible to apply a vacuum to the vessel containing the liquid egg product, and then optionally to break the vacuum with an inert gas and repeat this operation until bubbles cease to form at the surface of the product, while constantly checking the dissolved oxygen content. A vacuum of less than 80, and preferably 40 to 60 mm Hg is generally used. According to a preferred embodiment, the inert gas is bubbled through the liquid egg product in a controllable manner and an adjustable vacuum is maintained in the atmosphere above

the liquid egg product, so that the bubbling can be started at atmospheric pressure and the pressure progressively reduced whilst decreasing the flow of inert gas until, at the end of the operation, a vacuum is obtained, the value of which is chosen according to the osmotic pressure of the product and the temperature at the end of the treatment.

Volumetric pumps, which can operate at relatively low pressures, can be used for this purpose; the liquid egg is sprayed in an enclosure under a controlled vacuum and sucked continuously into the lower part of this enclosure in order to be led thereafter into a container or containers for preservation in the absence of air.

When it is desired to obtain a final product which is both concentrated and deoxygenated, it is also advantageous to combine the use of an ultrafiltration device with a degassing device.

Nitrogen, nitrous oxide, rare gases such as argon, "Freon" (Registered Trade Mark) 114 (1-chloro-1,1,2,2,2-pentafluoroethane) or other halogenated hydrocarbons are suitable inert gases.

When the degassing is effected by simple heating, the process may be carried out in the open air or in a sealed vessel in a stream of inert gas, with or without stirring. The egg product is heated preferably to 50 to 60° C for a time insufficient to cause denaturation of the proteins and of the fatty substances as well as coagulation, checking the oxygen content until it is less than 3 ppm.

Thus, the maximum heating for pure egg is generally 50° C for less than 6 hours. The maximum heating for a concentrated egg product which contains 50% by weight of sugar is generally 75° C for less than 4 hours. However, good results can be obtained with heating times from twenty minutes to a few hours.

The heating is then stopped and the product allowed to cool, or it is cooled in heat exchangers.

In the case of fragile products, particularly pure egg whites into which the additives have previously been introduced, the deoxygenation is advantageously achieved both by entraining with a neutral gas and by heating.

It is also advantageous to carry out the deoxygenation, particularly in the case of deoxygenation by simple heating, with a tubular installation in which the egg product circulates, it being possible for the temperature variations to be easily obtained by jacketing with a heating or cooling fluid, the circulation of which is adjusted at will.

After stages (a) and (b), the deoxygenated liquid egg product should be preserved in the absence of air, preferably under an inert atmosphere, especially other than carbon dioxide, prior to stage (c) or packaging for consumption.

Stage (c) of the process of the invention consists in relatively prolonged heating the deoxygenated liquid egg product at a temperature which is lower than the coagulation temperature, for example 75° C for 4 hours or 50 to 65° C for several days, in a closed container or under an inert gas atmosphere. The treatment is carried out such as to prevent denaturation of the proteins and of the fatty substances, as well as coagulation.

We have found that excellent preservation of the egg product, approaching that of "sterilisation," can thus be obtained without causing denaturation of the proteins of the egg product. In this context, "sterilisation" is to be understood as meaning that a product is obtained which contains less than 1,000 microorganism germs per gram.

In particular, and contrary to the case of certain poorly sterilised preserved foodstuffs, enzymatic reactions which detract from the good preservation of the product are not observed when the egg products obtained after stages (a) and (b) are heated. Even better, after storage at temperatures from 45 to 65° C, the products of the invention become absolutely sterile in a few hours to a few days.

It is believed that the heating in stage (c) accelerates the phenomenon of auto-sterilisation of the liquid egg; the very small amount of residual oxygen and carbon dioxide gas and the increase in the osmotic pressure with temperature favour and accelerate the bactericidal enzymatic reactions of certain proteins in the egg.

The heating in stage (c) can be carried out either in customary sealed cells, under an atmosphere of, or with circulating, neutral gas, or in a tubular installation, or by heating the product obtained after stages (a) and (b) placed directly in its final hermetically-sealed unit packaging for commercial use.

It is advantageous, for certain products, to end the heat treatment of stage (c) with rapid cooling, which is easily obtained in the tubular installations or when the product is packaged after stages (a) and (b).

The temperature and duration of stage (c) can be determined for each of the egg products obtained after stages (a) and (b); the concentration, additives content and oxygen content in ppm, as well as the initial degree of biological purity, can be checked against previously prepared tables and charts.

A sterilised liquid egg product according to the invention can thus be obtained, in which the number of microorganism germs per gram can be lower than 1,000.

The product can be placed in its final hermetic packaging after stages (a) and (b) of the process, or stored at ambient temperature; it should always be kept under neutral atmosphere prior to stage (c). When in the final hermetic packaging the product may be stored at ambient temperature.

The various stages of the process of the

invention can be carried out continuously or discontinuously.

Stages (a) and (b) essentially comprise the checking of the removal of oxygen to a content which does not exceed 3 ppm without denaturation of the proteins and fatty substances, as is the case, moreover, in the optional heating stages (c). In the last case, the oxygen content can be only checked after stage (c), the oxygen content always being less than 3 ppm. Denaturation is well known and can be caused by, for example, treatment at too high a temperature or the addition of too much salt or sugar.

The analytical and organoleptic qualities of the end products, which qualities can be checked for each of the uses of the products, can be used to prepare the tables and charts referred to above.

The process of the invention makes it possible to prevent the denaturation of the proteins such as lysozyme, ovotransferrin, ovomucoid, ovomucin, and ovoinhibitors which are originally contained in the egg and which have a direct or indirect action on the micro-organisms both as regards their survival and their multiplication, or also with regard to the blocking of the microbial enzymes. It is therefore of the greatest significance that the proteins remain relatively unchanged, in the process of the invention.

The liquid egg product which is obtained after stages (a) and (b), contains less than 3 and preferably less than 1 ppm of oxygen and, after 15 days at 20° C, generally less than 15,000 and preferably less than 1,500 germs per gram. This product can be stored for several months whilst retaining excellent organoleptic properties; it can be used in a completely analogous manner to the corresponding fresh products, and can even show an improvement in certain cases, such as good preservation of a whipped product.

We have verified, for example, that the white of "gathered" eggs, treated according to the process of the invention, exhibits new and improved characteristics even relative to egg white coming from freshly laid unbroken eggs for which the average dissolved oxygen content is 3.4 ppm and the average pH is 9 to 9.2 for 2 to 3 day old eggs.

A product based on egg white generally contains either at least 9% by weight of salt, or at least 45% of sugar, or both salt and sugar with an osmotic pressure equal to at least 20 atmospheres, the pH being from 8.60 to 8.85. A product based on egg yolk generally contains either at least 5% by weight of salt, or at least 40% of sugar, or both salt and sugar with an osmotic pressure equal to at least 20 atmospheres, the pH being from 6.30 to 6.45. A product based on whole egg, or egg which is reconstituted in variable proportions, generally contains either at least 5% and preferably 9% of salt, or at least 40% and preferably 45% of sugar, or both salt and sugar with an osmotic pressure equal to at least 20 atmospheres, the pH being from 6.30 to 8.85.

After stage (c) the product generally contains less than 5,000 and preferably less than 1,000 germs per gram (at the time of storage). This product can be stored for several months whilst retaining all its organileptic properties.

Relative to the degassed egg product obtained after stages (a) and (b) of the process, the egg product obtained after heating according to stage (c) has the advantage, for the same period of preservation, of generally having a lower osmotic pressure, that is to say of containing smaller proportions of foodstuff ingredients such as salt and sugar; this is desirable for certain uses.

The properties of the product impart remarkable bacteriological stability and a high degree of safety for very varied uses, even if, in certain cases, the product is exposed to the air for several weeks before its consumption. The duration of this stability is of course dependent on the osmotic pressure of the product, it being possible for the preservation of the sterilised egg product of the invention to be as much as 6 to 8 months if stage (c) is carried out. The products can be used in food for human consumption or in any other application.

When stage (c) has been carried out the product based on egg whites, generally contains either at least 5% and preferably 7% by weight of salt, or at least 30% and preferably 35% of sugar, or salt and sugar with an osmotic pressure equal to at least 20 atmospheres, and less than 5,000 and preferably less than 1,000 germs per gram. The corresponding product based on egg yolks, generally contains either at least 5% and preferably 7% by weight of salt, or at least 35% and preferably 40% of sugar, or both salt and sugar with an osmotic pressure equal to at least 20 atmospheres, and less than 2,000 and preferably less than 500 germs per gram. The corresponding product based on eggs which are whole or reconstituted in variable proportions, generally contains either at least 5% of salt, or at least 30% and preferably 35% of sugar, or both salt and sugar with an osmotic pressure equal to at least 20 atmospheres, and less than 2,000 and preferably less than 500 germs per gram.

Furthermore, if freshly cracked eggs contain a large number of germs, it can be advantageous, without increasing the osmotic pressure of the product, to follow stages (a) and (b) of the process with the heating stage (c) and thus ensure better preservation of the product.

Relative to similar liquid egg products, it has been found that the products of the invention exhibit improved properties with

regard to swelling, particularly in meringues and ice-creams, g od coagulation of the proteins in confectionery, particularly for Angel-cakes, and emulsifying properties, particularly in mayonnaises. In particular, the egg products of the invention give much better results than the conventional pasteurised egg products, with regard to swelling.

The following Examples, in which "gathered" hens' eggs of the usual quality have been used, further illustrate the present invention. "Gathered" eggs are eggs which have been laid 8 to 30 days before being cracked and treated. Except where indicated to the contrary, the temperatures are in degrees Centigrade.

EXAMPLE A.

A 200 g sample of well mixed liquid egg yolks is prepared, which has a solids content of 43%, a pH of 6.54, and which contains 190,000 germs per gram. 100 g are removed to form the sample A_1 which, after having taken a 10 g sample of product for analysis of the dissolved oxygen, is preserved at 20° C in an enclosure which is hermetically sealed and under a nitrogen atmosphere.

A sample A_2, previously homogenised and containing 50 g of egg yolk coming from the same initial mixture, 48 g of sugar and 2 g of salt, is placed in a sealed enclosure to which a vacuum is applied down to a pressure of 50 mm Hg by means of a P. PIEL MARC 702 pump, and this pressure is maintained for 15 seconds. The vacuum is broken with nitrogen and these operations are repeated until foam no longer forms at the surface of the product. All the abovementioned operations are carried out at ambient temperature.

A 10 g sample of the mixture is taken for analysis of the dissolved oxygen and the remainder of the product is preserved under the same conditions as for A_1.

The oxygen analyses are carried out immediately for A_1 and A_2 by means of a YSI 54 dissolved oxygen analyser (Yellow Spring Instrument, Yellow Springs Ohio 45387 USA). The total flora in sample A_2 is determined after 1 month.

The results are as follows:

	dissolved oxygen	total flora determination impossible
Sample A_1	8.7 ppm	(sample destroyed)
Sample A_2	2.7 ppm	1,600 germs/g.

Analogous good results are obtained by proceeding in the same manner as above but without breaking the vacuum to the extent that the formation of foam is prevented. Preferably, the treatment is then completed by applying a vacuum of a few mm Hg for more than 10 minutes.

EXAMPLE B.

A series of preservation tests on liquid egg yolks is carried out starting from 10 kg of perfectly mixed liquid egg yolks, of which a 200 g sample is taken for each of the tests with a view to the preparation of the egg products according to the invention, and the initial characteristics of which are as follows:

Solids content: 47% by weight
pH: 6.58
Viscosity: 440 centipoises (cP) measured with a unit number 4 or 3 PROLABO type DRAGE viscometer.
Dissolved O_2: 7 ppm (measured as indicated in Example A)
CO_2 capable of being liberated: 1.5 millimol per millilitre (measured with a "CO_2 apparatus SET" manufactured by HARLECO HERSTAL)
Total flora: 980 germs per gram (or germs/g.).

The 200 g sample is made up with the amounts of salt (sodium chloride of foodstuff quality), sugar (sucrose) or both salt and sucrose as indicated in percentage by weight in Table I below. After perfect homogenisation at a temperature of 20° C in a round-bottomed flask equipped with a stirrer, 150 g samples of the mixture are taken each time and are placed in another round-bottomed flask equipped with a bubbler for the introduction of gas, and with a thermometer. In each test, the temperature is adjusted to 20° C by means of a water-bath and nitrogen is bubbled for 10 seconds at a flow-rate of 3 litres per minute. At the end of each test, the viscosity, the pH, the amount of dissolved oxygen and the amount of carbon dioxide gas which is capable of being liberated are measured as indicated above, and the osmotic pressure and the corresponding solids content are determined.

100 g samples of each product obtained after bubbling are taken and they are placed in a hermetically sealed enclosure at a temperature of 20° C. The total flora is determined for each sample after 15 days.

Sample B_1, which contains neither salt nor sugar, is found to be destroyed after 5 days. On the other hand, samples B_2 to B_8, which contain 5 to 15% of salt respectively, are perfectly preserved.

With regard to the samples which only contain sugar, only samples B_{12} to B_{15}, that is to say those which contain more than 40% of sugar, exhibit good preservation.

In the case of the samples which contain both salt and sugar, it is found that the salt content should preferably be at least 0.5% and the sugar content at least 47.5%, which corresponds to samples B_{19}, B_{20} and B_{21}. The above tests make it possible to determine the lower limits for the percentages of salt and

sugar when the dissolved oxygen content is of the order of 2 to 2.3 ppm. However, it was of value to know if the acceptable light might not be higher. This limit was sought for a product based on liquid egg yolks which was prepared under the conditions in Table I below and contained 47.5% of sugar, but this was carried out on samples for which the bubbling time varied from 0 (before bubbling) to 10 seconds. Tests B_{24} and B_{25} show that the upper acceptable limit for residual dissolved oxygen in the liquid egg yolk treated according to the process of the invention is of the order of 2 to 3 ppm. All the abovementioned tests are carried out at ambient temperature.

Analogous results are obtained by carrying out a first degassing before the introduction of the salt and/or sugar, but in this case, it is necessary to end the treatment with a further degassing whilst checking the dissolved oxygen content as indicated above.

EXAMPLE C.

A series of preservation tests on liquid egg whites is carried out starting from 10 kg of non-concentrated, perfectly mixed liquid egg whites, of which 200 g samples are taken for each of the tests with a view to the preparation of the egg product according to the invention, and the initial characteristics of which are as follows:

Solids content: 12%
pH: 9.2
Viscosity: 5 cP (unit number 2 PROLABO viscometer)
Dissolved O_2: 10 ppm
CO_2 which is capable of being liberated: 39 millimols per millilitre.
Total flora: 12,500 germs/g.

All measurements are carried out as in Example B and the procedure followed is furthermore exactly as in this Example B.

The results are indicated in Table II below.

Sample C_1, which contains neither salt nor sugar, is found to have a flora of more than 100,000,000 germs/g after 15 days at 20° C. In the samples only containing salt, the proportion of salt is preferably equal to at least 9% by weight as indicated in test C_4 which has a flora of 5,000 germs/g. With regard to the samples only containing sugar, the proportion of sugar is preferably equal to at least about 40% by weight as in C_{12}. In the case of the samples which contain both salt and sugar, it is necessary for the proportion of salt to be at least 0.5% by weight and the proportion of sugar at least 47.5% by weight as in C_{19}.

As for Example B, the upper acceptable limit for residual dissolved oxygen in the liquid egg white treated according to the process of the invention is again of the order of 2 to 2.5 ppm as indicated by samples C_{24} and C_{25}.

In Examples A, B and C, argon, nitrous oxide, Freon 114 or Freon 115, with a treatment temperature of less than 50° C and preferably 10 to 35° C, can be used with comparably good results.

Analogous good results are obtained with egg products which are concentrated to a percentage solids content which does not exceed 60%; this also applies to egg yolks and egg whites as well as to whole eggs, the proportions of salt or sugar furthermore remaining unchanged.

EXAMPLE D.

400 g of concentrated whole egg having a solids content of 48% are prepared. 50 g are removed to form the sample D_1 which, after having taken a 10 g sample of product for analysis of the dissolved oxygen, is preserved at 20° C in hermetically sealed enclosures and under a nitrogen atmosphere.

A 200 g sample of the initial concentrated whole egg mixture is taken, to which 200 g of sucrose are added; after the whole mixture has been well mixed, samples D_2 and D_3, each of 100 g, are taken.

Sample D_2 is heated in a water bath for 15 minutes at 50° C and sample D_3 for 15 minutes at 65° C, and the samples thus treated are then allowed to cool.

The oxygen content and the number of germs per gram are determined as in Example A for the samples which have been preserved for 15 days at a temperature of 20° C and then preserved for 72 hours at a temperature of 30° C. The results are as follows:

	Dissolved oxygen in ppm	Number of germs per g destroyed
Sample D_1	4	
Sample D_2	1.5	900
Sample D_3	0.3	150

Analogous results are obtained with whole egg whatever the solids content may be.

EXAMPLE E.

A series of preservation tests on liquid egg white is carried out starting from 20 kg of non-concentrated, perfectly mixed liquid egg white having a solids content of 11% by weight.

100 g are removed, to form the sample E and, after having taken a 10 g sample of product for analysis of the dissolved oxygen, it is preserved at 20° C in a hermetically sealed enclosure and under a nitrogen atmosphere.

The following samples are then taken; a sample E_2 of 100 g, a sample E_3 of 90 g, and a sample E_4 of 50 g to which 50 g of sucrose are added and which is perfectly mixed.

Samples E_2, E_3 and E_4 are heated in a water bath at a temperature of 50° C for 20 minutes and the heating is stopped. 10 g of salt are added to sample E_3 and perfectly mixed with the egg white.

A 10 g sample is immediately taken for analysis of the dissolved oxygen and the remainder of each sample is placed in a hermetically sealed enclosure at a temperature of 20° C for a determination of the total flora after 15 days of preservation, as in the preceding examples.

The following results are obtained:

	Dissolved oxygen in ppm	Number of germs per g
Sample E_1	7	6000,000
Sample E_2	2	4500,000
Sample E_3	1.5	1,200
Sample E_4	1.3	950

EXAMPLE F.

A series of preservation tests on liquid egg yolks is carried out starting from egg yolks which have already undergone deoxygenation according to stages (a) and (b) of the process of the invention and which are subjected to the prolonged heat treatment according to stage (c) of the process.

Test F_1 is carried out starting from 10 kg of perfectly mixed liquid egg yolks having a solids content of 44% by weight and containing 380,000 micro-organism germs per gram.

A 200 g sample of egg yolk is taken, to which 200 g of sugar are added, whilst stirring until a perfectly homogeneous mixture is obtained. Deoxygenation is then carried out as indicated in Example B. The oxygen content as measured at the end of the deoxygenation, that is to say at the end of stages (a) and (b), is 2.9 ppm. The whole mixture is heated at a temperature of 65° C for 72 hours in a stream of nitrogen, and an egg product is obtained which has the following characteristics:

Solids content: 72.0%
Dissolved oxygen: 0.5 ppm (measured as indicated in Example A)
Number of germs per gram: less than 10.

The product is not coagulated and the proteins contained therein show practically no denaturation.

The procedure is carried out in exactly the same manner for tests F_2, F_3 and F_4 which are all carried out on egg yolks which have a solids content of 44%, but for which the conditions of the treatment have been changed as indicated in Table III below.

This table shows the improvement made by the heating stage (c) on the products which have already been subjected to deoxygenation according to stages (a) and (b).

In particular, the final egg product has a dissolved oxygen content of less than 1 ppm and a number of germs per gram which is lower than 500 and usually lower than 100.

Comparably good results are obtained after stage (c), when stages (a) and (b) are carried out in any order, and when deoxygenation is achieved either by applying a vacuum of the order of 80 mm Hg or by simple heating until an oxygen content of less than 3 ppm is obtained, as in Examples E and D, the heat treatment according to stage (c) then being carried out as in Examples F_1 to F_4.

EXAMPLE G.

A series of preservation tests on liquid egg whites is carried out starting from egg whites which have undergone deoxygenation according to stages (a) and (b) of the process of the invention and which have been subjected to the prolonged heat treatment according to stage (c) of the process.

Tests G_1 and G_2 are carried out starting from 10 kg of perfectly mixed, concentrated liquid egg whites which have a solids content of 33% by weight and contain 99,000 micro-organism germs per gram before concentration until a solids content of 33% is obtained. 200 g of the mixtures G_1 and G_2, which contain either 11% of sodium chloride or 50% of sugar respectively, are prepared and deoxygenation is carried out as indicated in Example B. The oxygen content as measured at the end of the deoxygenation is 2.7 and 2.5 ppm respectively for tests G_1 and G_2. The two samples are then heated to temperatures of 50 and 65° C respectively for 24 hours, and a final product is obtained, the characteristics of which are indicated in Table III.

The procedure is carried out in the same manner for tests G_3, G_4 and G_5, but starting from egg whites containing 90,000 micro-organism germs per gram.

Table III shows the particular conditions of these tests, especially with regard to the salt or sugar content and the temperature and duration of stage (c), and it also shows the oxygen content and the number of germs contained in the final product.

In this case, comparably good results are again obtained when the deoxygenation stage is carried out under a partial vacuum or by simple heating.

EXAMPLE H.

A series of preservation tests on whole eggs is carried out starting from concentrated whole eggs which have undergone deoxygenation according to stages (a) and (b) of the process of the invention and which are subjected to the prolonged heat treatment according to stage (c) of the process.

Tests H_1 to H_4 are carried out starting each time from 10 kg of perfectly mixed whole eggs, which are concentrated to a solids

content of 48% but in which the number of micro-organism germs is different, as indicated in Table III below. The tests are carried out on 200 g amounts of the respective mixtures as indicated in Table III, and deoxygenation is carried out as in Example B. Furthermore, Table III indicates for each test the conditions of the prolonged heating according to stage (c), as well as the characteristics of the products obtained after deoxygenation and those of the final product.

In this case, analogous results are again obtained for the final product when deoxygenation is carried out under a partial vacuum or by simple heating as in Examples D and E.

According to Example H_5, 1,000 kg of a mixture containing 665 kg of whole eggs concentrated to 48%, 330 kg of sugar and 5 kg of salt are heated, whilst stirring, at a temperature of 55° C for 72 hours in a sealed cell comprising a device for sweeping nitrogen. The initial number of germs in the whole egg before concentration was 950,000 germs per gram.

The check was not carried out after the deoxygenation stages (a) and (b). A final egg product is obtained which contains 260 germs per gram and the oxygen content of which is 0.9 ppm.

At the end of the treatment, the product is placed in unit packaging containing 200 g of product, still under a nitrogen atmosphere; these packagings are hermetically sealed and stored. A check carried out after 11 days of storage at ambient temperature indicated an average content of 100 germs per gram in the egg product.

As for products of egg yolks and egg whites, analogous results are obtained for products of whole eggs when deoxygenation is carried out under a partial vacuum or by simple heating as in the case of Example H_5.

Furthermore, we have observed that, in general, preservation of the products of Examples F, G and H at a temperature of 50° C for several months resulted in a tendency for the number of micro-organism germs to diminish; this is of considerable value for certain countries, especially for countries with a tropical climate.

Completey comparable results have been obtained by treating egg products concentrated to a solids content of 60% in an analogous manner to that of Example H.

TABLE I (egg yolks)

No. B_1–B_{25}	Ingredients		Bubbling time in seconds	Osmotic pressure in atmospheres	Viscosity in cP
	NaCl% by weight	Sugar % by weight			
1	0	0	10	0	440
2	5	0	10	38.3	1,450
3	7.5	0	10	57.5	2,800
4	9	0	10	69	4,750
5	10	0	10	77	6,000
6	11	0	10	84.3	8,300
7	12	0	10	92	10,000
8	15	0	10	115	22,600
9	0	10	10	6.5	200
10	0	20	10	13	300
11	0	30	10	19.6	500
12	0	40	10	26	620
13	0	45	10	29.4	800
14	0	47.5	10	31	900
15	0	50	10	32.6	3,500
16	0.5	45	10	33.2	800
17	1	45	10	37.1	900
18	2	45	10	44.8	800
19	0.5	47.5	10	34.8	1,340
20	1	47.5	10	38	2,600
21	2	47.5	10	45	2,500
22	0	47.5	0		
23	0	47.5	5		
24	0	47.5	8		
25	0	47.5	10		

TABLE I (egg yolks) Continued/ ....

No. B_1–B_{25}	Solids Content % by weight	pH	Dissolved gases		MESOPHILE AEROBIC FLORA (in number of germs per gram after 72 hours at 30°C on samples preserved for 15 days at 20°C)
			O_2 ppm	CO_2 mmol	
1	47	6.58	2.5	1	Sample decayed after 5 days
2	49.6	6.38	2.3	1	350
3	51	6.36	2.3	1	50
4	51.8	6.32	2.2	1	50
5	52.3	6.30	2.3	1	50
6	52.8	6.27	2.3	1	30
7	53.4	6.25	2.2	1	–
8	55	6.25	2.1	1	–
9	52.3	6.54	2.2	1	86,000,000
10	57.6	6.52	2.3	1	26,250,000
11	62.9	6.49	2.1	1	13,200,000
12	68.2	6.45	2.2	1	1,550
13	70.8	6.47	2.2	1	570
14	72.1	6.44	2.3	1	300
15	73.5	6.62	2.1	1	40
16	71.1	6.40	2.1	1	540
17	71.3	6.35	2.2	1	1,820
18	72	6.33	2	1	5,600
19	72.4	6.43	2	1	1,560
20	72.7	6.36	2.1	1	115
21	73.2	6.33	2	1	205
22			8	1.5	30,000
23			5	1	20,000
24			3	1	1,000
25			2	1	300

TABLE II (egg whites)

No. C_1–C_{25}	Ingredients		Bubbling time in seconds	Osmotic pressure in atmospheres	Viscosity in cP
	NaCl% by weight	Sugar % by weight			
1	0	0	10	0	5
2	5	0	10	38.3	20
3	7.5	0	10	57.5	25
4	9	0	10	69	27
5	10	0	10	77	30
6	11	0	10	84.3	35
7	12	0	10	92	35
8	15	0	10	115	35
9	0	10	10	6.5	45
10	0	20	10	13	45
11	0	30	10	19.6	60
12	0	40	10	26	100
13	0	45	10	29.4	190
14	0	47.5	10	31	190
15	0	50	10	32.6	240
16	0.5	45	10	33.2	160
17	1	45	10	37.1	140
18	2	45	10	44.8	140
19	0.5	47.5	10	34.8	180
20	1	47.5	10	38	180
21	2	47.5	10	45	170
22	0	47.5	0		
23	0	47.5	5		
24	0	47.5	8		
25	0	47.5	10		

WHAT WE CLAIM IS:—

1. Process for the preservation of a liquid egg product which comprises:

(a) adding homogeneously either salt in an amount of at least 5% by weight, or sugar in an amount of at least 30% by weight, based on the weight of the final product or both salt and sugar in amounts such that the osmotic pressure of the resulting mixture is at least 20 atmospheres, to a concentrated or un-concentrated liquid egg product;

(b) removing dissolved gases from the liquid egg product until the oxygen content is less than 3 ppm based on the weight of the final product, stages (a) and (b) being carried out in either order;

(c) optionally heating the product obtained after stages (a) and (b) at a temperature below the coagulation temperature; and

(d) optionally placing the product in an air-tight container under reduced pressure or in the presence of a gas which is neutral to foodstuffs, other than carbon dioxide gas, the temperature never exceeding the coagulation temperature of the egg product during the process.

2. Process according to claim 1 in which stage (b) is carried out after stage (a).

3. Process according to claim 1 in which stage (b) is carried out before stage (a).

4. Process according to any one of claims 1 to 3 in which stage (b) is carried out by bubbling through a gas which is inert to foodstuffs.

5. Process according to claim 4 in which nitrogen, argon, 1-chloro-1,1,2,2,2-pentafluoroethane, or nitrous oxide is used as the gas.

6. Process according to claim 4 or 5 in which the bubbling is carried out in a closed circuit, and, after bubbling, the gas is purged of the carbon dioxide gas and/or the oxygen contained therein, before being bubbled through the liquid egg product again.

7. Process according to any one of claims 1 to 3 in which stage (b) is carried out by heating at a temperature of 45 to 75° C for a time insufficient to cause denaturation of the proteins or coagulation of the product.

8. Process according to claim 7 in which the said temperature is 50° to 65° C.

9. Process according to any one of claims 1 to 3 in which stage (b) is carried out by subjecting the product to a pressure not exceeding 80 mm Hg.

10. Process according to claim 9 in which the said pressure is 40 to 60 mm Hg.

11. Process according to any one of claims 1 and 3 to 10, in which stage (b) is carried out on a previously concentrated product before stage (a).

12. Process according to claim 11 in which the concentration is carried out by ultrafiltration.

13. Process according to claim 12 in which degassing and the ultrafiltration are carried out simultaneously.

14. Process according to any one of the preceding claims in which salt is added in an amount of at least 7% by weight or sugar is added in an amount of at least 35% by weight or a mixture of salt and sugar is added to provide an osmotic pressure in the product of 25 atmospheres.

15. Process according to any one of the preceding claims in which the liquid egg product is egg yolk.

16. Process according to claim 15 in which stage (d) but not stage (c) is carried out and either salt in an amount of at least 5% or sugar in an amount of at least 40% or a mixture of salt and sugar is added in stage (a).

17. Process according to claim 15 in which stage (b) is carried out as defined in claim 7 or 8 and either salt in an amount of at least 5% or sugar in an amount of at least 35% or a mixture of salt and sugar is added in stage (a).

18. Process according to claim 17 in which the salt is added in an amount of about 7% or sugar is added in an amount of about 4%.

19. Process according to any one of claims 1 to 14 in which the liquid egg product is egg white.

20. Process according to claim 19 in which stage (d) but not stage (c) is carried out and either salt in an amount of at least 9% or sugar in an amount of at least 35% or a mixture of salt and sugar is added in stage (a).

21. Process according to claim 20 in which the salt is added in an amount of about 7% or the sugar is added in an amount of about 45%.

22. Process according to claim 19 in which stage (b) is carried out as defined in claim 7 or 8 and either salt in an amount of at least 5% or sugar in an amount of at least 30% or a mixture of salt and sugar is added in stage (a).

23. Process according to claim 22 in which the salt is added in an amount of about 7% or the sugar is added in an amount of about 35%.

24. Process according to any one of claims 1 to 14 in which the liquid egg product is whole egg.

25. Process according to claim 24 in which stage (d) but not stage (c) is carried out and either salt in an amount of at least 5% or sugar in an amount of at least 40% or a mixture of salt and sugar is added in stage (a).

26. Process according to claim 25 in which the salt is added in an amount of about 9% or the sugar is added in an amount of about 45%.

27. Process according to claim 24 in which stage (b) is carried out as defined in claim

7 or 8 and either salt in an amount of at least 5% or sugar in an amount of at least 30% or a mixture of salt and sugar is added in stage (a).

28. Process according to claim 27 in which the sugar is added in an amount of about 35%.

29. Process according to any one of the preceding claims in which in stage (b) the oxygen content is reduced to below 1 ppm based on the weight of the final product.

30. Process according to claim 1 substantially as hereinbefore described.

31. Liquid egg product whenever obtained by a process as claimed in any one of the preceding claims.

32. A liquid egg product according to claim 31 which is packaged such that at the time of packing the number of microorganisms per gram is less than 15,000.

33. A liquid egg product according to claim 32 which at the time of packing contains less than 1,500 microorganisms per gram.

34. A liquid egg product according to claim 31 prepared by a process as claimed in any one of claims 17, 18, 22, 23, 27 and 28 which is packaged such that at the time of packing its oxygen content is less than 3 ppm and the number of microorganism germs per gram is less than 5,000.

35. A liquid egg product according to claim 34 which at the time of packing contains less than 1,000 microorganisms per gram.

36. A packaged liquid egg product based on the yolks of hens' eggs and containing either at least 5% by weight of salt, or at least 40% by weight of sugar, or salt and sugar in amounts such that the osmotic pressure of the product is at least 20 atmospheres, and having, at the time of packaging, an oxygen content of less than 3 ppm, and a microorganism content of less than 15,000 germs per gram.

37. A packaged liquid egg product based on the whites of hens' eggs and containing either at least 9% by weight of salt, or at least 35% by weight of sugar, or both salt and sugar in amounts such that the osmotic pressure of the product is at least 20 atmospheres, and having, at the time of packaging, an oxygen content of less than 3 ppm, and a microorganism content of less than 15,000 germs per gram.

38. A liquid egg product according to claim 37 which contains about 45% by weight of sugar.

39. A liquid egg product based on whole hens' eggs, and containing either at least 5% by weight of salt, or at least 40% by weight of sugar, or salt and sugar in amounts such that the smotic pressure of the product is at least 20 atmospheres, and having an oxygen content of less than 3 ppm, and a microorganism content of less than 15,000 germs per gram.

40. A liquid egg product according to claim 39 which contains about 9% by weight of salt or about 45% by weight of sugar.

41. A liquid egg product according to any one of claims 36 to 40 having a microorganism content of less than 1,500 germs per gram.

42. A liquid egg product based on yolks of hens' eggs and containing either at least 5% by weight of salt, or at least 35% by weight of sugar, or salt and sugar in amounts such that the osmotic pressure of the product is at least 20 atmospheres, and having an oxygen content of less than 3 ppm, and a microorganism content of less than 5,000 germs per gram.

43. A liquid egg product according to claim 42 which contains about 7% by weight of salt or 40% by weight of sugar.

44. A liquid egg product based on whites of hens' eggs, and containing either at least 5% by weight of salt, or at least 30% by weight of sugar, or both salt and sugar in amounts such that the osmotic pressure of the product is at least 20 atmospheres, and having an oxygen content of less than 3 ppm, and a microorganism content of less than 5,000 germs per gram.

45. A liquid egg product according to claim 44 which contains about 7% by weight of salt or about 35% by weight of sugar.

46. A liquid egg product based on whole hens' eggs, and containing either at least 5% by weight of salt, or at least 30% by weight of sugar, or salt and sugar in amounts such that the osmotic pressure of the product is at least 20 atmospheres, and has an oxygen content of less than 3 ppm, and a microorganism content of less than 5,000 germs per gram.

47. A liquid egg product according to claim 46 which contains about 7% by weight of salt or about 35% by weight of sugar.

48. A liquid egg product according to any one of claims 42 to 47 having a microorganism content of less than 1,000 germs per gram.

49. A liquid egg product according to any one of claims 36 to 48 which contains salt and sugar such that the osmotic pressure is about 25 atmospheres.

50. A liquid egg product according to any one of claims 36 to 49 having an oxygen content of less than 1 ppm.

J. A. KEMP & CO.,
Chartered Patent Agents,
14, South Square,
Gray's Inn, London, WC1R 5EU.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1979.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.